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14. ABSTRACT

The results of a three-year study of attempts to produce magnetically soft materials by mechanical milling show that a major obstacle to the achievement of the desired results of high saturation magnetization with low magnetic anisotropy, using iron-cobalt, is the extreme hardness of the particles after milling. This is the compaction problem that must be solved for the material to be useful for technical applications. The project did result in the establishment of three research facilities that have subsequently led to major new insights into micromagnetics, magnetic random access memory, and the magnetic structure of Fe(Al) alloys.

This project led to additional funding of research at Virginia State University, an Historically Black College/University, and has stimulated interest among students in physics research, subsequently leading them to seek advanced degrees in physics.

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Final Report

Microscopic Characterization of Novel Magnetic Materials With Potential for Major Technological Impact

Physics Department
Virginia State University
Petersburg, VA 23806

For the period
September 16, 1997 –July 31, 2000

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Carey E. Stronach, Ph.D.
Project Director

Anthony S. Arrott, Ph. D.
Scientific Director

In the fall of 1996, Virginia State University requested funding for an experimental and theoretical study of iron-based materials produced by mechanical milling and mechanical alloying. The choice of iron-based materials was made because iron is the basis of much of structural technology, the electromagnetic generation of power, and the storage of information, all of which should have applications of interest to the Ballistic Missile Defense Organization. Funds were granted for two years, after which a one-year supplement and extension was applied for and obtained.

The principal investigators, Professor Carey E. Stronach and Dr. David R. Noakes brought to the project their wide experience in muon spin rotation measurements on many materials, quite a few of which were magnetic. They had for many years carried out their research program using the facilities at TRIUMF (TRI-University Meson Facility in Vancouver), bringing with them Virginia State University students to experience world-class research on contemporary problems in physics. Having seen the possibility of support from the Department of Defense, they enlisted the aid of Professor Anthony S. Arrott in devising a program that should interest the Air Force Office of Scientific Research and bring Arrott to Virginia State University if funded.

They proposed that a ball-milling laboratory be established at Virginia State University, and that the materials produced with this technique be characterized using neutron scattering and muon spin rotation. Among the materials suggested for study were the iron nitrides and alloys of iron with aluminum.

Early in the project the goal became the achievement of a soft magnetic material in laminated form for use in high-temperature motors requiring high saturation magnetization and good mechanical stability. This was stimulated by the Air Force desire for a "more electric" airplane. The research was to be guided by the computational methods of micromagnetics and a background of metallurgical experience. Alloys of iron with cobalt Fe(Co) were chosen because of the possibility that the alloy with the highest saturation magnetization could be made magnetically soft through exchange softening of the magnetic anisotropy, which occurs in very fine-grained materials. Mechanical milling produces the necessary fine grains of less than 10-nm size in particles that typically have dimensions from 10 to 100 μm. The particles then must be compacted to form full density laminates for magnetic applications.

Unfortunately, the use of mechanical milling to achieve the necessary fine grain size in Fe(Co) produces particles that are mechanically extremely hard. They are so hard that almost no compaction is experienced when pressed to the limit of catastrophic failure of the compression dies. This is the "compaction problem" that must be solved if the promise of using Fe(Co) is to be realized. Though the full objective of the research has been so far thwarted, many positive results have been achieved along the way. The extreme hardness of these particles might in itself be of technological importance, but this possibility has not been pursued at Virginia State University.

The foremost accomplishment during this project was the establishment of three research laboratories at Virginia State University. The first of these contains the mechanical milling equipment, the second is the physical measurements room and the third is the laboratory for computations in micromagnetics. The use of external facilities was expanded to include access to analysis equipment in the Metallurgy division of NIST and the neutron diffractometer at the Missouri University Research Reactor, while maintaining the use of the muon spin relaxation beam line at TRIUMF.

The original proposal called for the purchase of the Australian built Uni-ball mill. A quote for this was included in the proposal, but after receiving the funding, it was found that the manufacturer would not deliver it. An alternative was found by adapting a puck-and-saucer rock crusher to mechanical milling. This had the advantages of producing orders of magnitude more material than conventional ball mills, but had the disadvantages of not having been engineered for that purpose. Much of the early effort, after the one year delay caused by the change in plans, went into developing the rock crusher into a laboratory tool that could sustain a positive pressure of argon and be cooled by water during the hours needed for milling as opposed to the minutes needed for rock crushing. A vacuum desiccant system, which was large enough to handle the 33 cm diameter saucer, was obtained on the used equipment market. This made it possible to work with pyrophoric powders. Furnaces suitable for annealing of the powders were obtained, also on the used equipment market. Vacuum facilities were already in place at Virginia State University in the form of a Varian turbo pump station. The mechanical milling laboratory was established in the old glass shop, which made it convenient for handling the pyrex capsules for heat treatment of the powders produced. Basic machine shop equipment was acquired for use in this laboratory.

Since the end of this project, the mechanical milling laboratory has produced materials of significant interest, particularly in the studies of Fe(Al) alloys that grew out of the work on Fe(Co) alloys. The original proposal discussed the possibility of the phenomenon of inverse melting. This was eventually discovered at Virginia State University in Fe(Al) alloys.

By the end of the project, the measurement laboratory was coming into being. The first equipment was a Perkin-Elmer Differential Scanning Calorimeter. This became the main tool for studying the thermal properties of the powders of Fe(Co) produced using the rock crusher. An electromagnet with 15-inch pole faces and the accompanying power supply and chiller was acquired for magnetic measurements from the Honeywell Corporation, at no cost to the project. Unfortunately delays in modifying laboratory space suitable to hold the large magnet made the magnet unavailable until the very end of the period of this grant. During that time, experiments in developing a new method of magnetic measurement were carried out in Prof. Arrott's laboratory in Vancouver. These were eventually transferred to the measurements laboratory at Virginia State University. During the course of this project major funding was obtained for the development of the measurement laboratory, but most of the equipment was not delivered until after the end of the project. The present well-equipped laboratory had its start from this research project supported by the BMDO.

The theoretical work was begun using the LLG Micromagnetics Simulator program obtained by Prof. Arrott from other funds. What was at the end of this project a laboratory with only one computer, now consists of 11 computers running many problems related to exchange softening and to the use of patterned media for magnetic random access memories. This BMDO grant led to the creation of the Center for Interactive Micromagnetics at Virginia State University.

The work on Fe(Co) alloys required the use of x-ray facilities and equipment for magnetization measurements. Arrott became a Guest Scientist at NIST with full access to the laboratory of the Magnetic Materials Group headed by Dr. Robert D. Shull.

The involvement of students in the laboratory began with the arrival of the differential scanning calorimeter, which resulted in the senior thesis of Larry C. Harris, who is now a graduate student at Clemson University in astrophysics.

The practice of taking students to TRIUMF to obtain data and then returning to Virginia State University to analyze the results was continued during this time. Dr. Noakes supervised M.S. theses by Ceasar U. Jackson, Jr. and Morris F. White, Jr. on μSR in magnetic materials.

Dr. Noakes continued his studies of magnetic materials using μSR, from which many publications flowed. He completed his definitive work, a 400-page manuscript on "μSR studies of rare earth and actinide magnetic materials", published in the Handbook on the Physics and Chemistry of Rare Earths, Vol 32 (Elsevier, 2001). Additional works relevant to this project include "Monte Carlo simulation of general cases of static muon spin relaxation in disordered magnetic materials: long range magnetic order in alloys", "Magnetism of Frustrated RFe_6Al_6 compounds studied by μSR and Mössbauer Spectrometry", "Cauchy magnetic field component and magnitude distribution studies by the zero-field muon spin relaxation technique", "Magnetism of crystalline and nanostructured $ZnFe_2O_4$.", and papers on the magnetic properties of $GdMn_2$, YMn, CeCuSn, CePtSn, and $TbFe_6Al_6$.

Likewise, Prof. Stronach continued his participation in studies of the interplay of superconductivity and magnetic ordering, which culminated in an article in *Science*, "Anomalous Weak Magnetism in Superconducting $YBa_2Cu_3O_{6+x}$", Volume 292, June 1, 2001, plus several additional publications.

At the 4th International conference on Nanostructured Materials, Stockholm, June 1998, Prof. Arrott gave an invited talk entitled: "Production of Magnetically Soft Materials by Mechanical Milling?" This gave the rationale for the studies undertaken in this grant from the Ballistic Missile Defense Organization. At that time there had been no applications of mechanical milling that had succeeded in the production of soft magnetic materials for motors, generators and transformers. The question mark in the title reflected the uncertainties to be faced in trying to achieve this desirable aim. These uncertainties included lack of understanding of the role of grain boundaries when the volume of grain

boundaries is comparable to the volume of the grains. This led to the idea of studying Fe(Al) in addition to Fe(Co), because in the former, it should be possible to make the grain boundaries magnetic while keeping the grains non-magnetic, thus allowing a unique insight in this problem. The studies of Fe(Al) would eventually lead to the discovery by Noakes and Arrott of Spin Density Waves in iron-aluminides.